OPERATING INSTRUCTIONS
FOR THE
JANIS RESEARCH
SUPERTRAN SYSTEM

Janis Research Company
2 Jewel Drive
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(978) 657-8750

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SECTION 1

SAFETY

1.1 SUMMARY

All safety pressure relief valves are installed to provide protection to the equipment and operating personnel. Do not tamper with any pressure relief valve.

During sample change, it is possible for the radiation shield to remain cold even after the sample has warmed to room temperature. Use gloves when handling a cold radiation shield to avoid low temperature burns.

During system shutdown, the transfer line leg will be extremely cold upon removal from the storage dewar. Do not touch the transfer line with bare hands. Always wear insulating gloves when handling the transfer line after use.

Do not bend the transfer line to a radius of less than 8 inches, to prevent possible damage to the inner line.

Do not close the flow regulator valve on the transfer line more than hand tight. Never use a wrench or any tool on the flow regulator knob, to prevent damage to the needle valve and seat.

SECTION 2

INTRODUCTION

2.1 GENERAL DESCRIPTION

The Janis Research Supertran (ST) System is a continuous flow research cryostat that can be used to perform a wide variety of experiments in the temperature range from 1.5 to 325 K (500 K or 700 K optional.) Liquid helium or nitrogen is continuously transferred through a high efficiency superinsulated line to a copper sample mount inside the cryostat vacuum jacket. A needle valve is incorporated in the transfer line, and is used to regulate the cryogen flow to the sample mount.

2.2 SYSTEM COMPONENTS

The Janis ST system includes the following components:

1. Cold finger cryostat: This is the part of the system that interfaces with samples. It includes a copper cold finger with tapped mounting holes, heater, and thermometer for monitoring and regulating the temperature. The system is designed to operate with either LN_2 or LHe as the working cryogen.

2. High efficiency transfer line: The ST transfer line combines vacuum insulation with multilayer superinsulation to provide low cryogen losses during transfer. A needle valve flow regulator is built-in to the transfer line. One end of the transfer line is inserted into the cryogen storage container, while the other end is inserted into the cold finger cryostat.

3. Radiation shield: This shield, usually fabricated from polished aluminum tubing, is mounted to a thermal anchor on the cold finger cryostat. It intercepts room temperature radiation, thereby reducing the heat load on the sample. This helps the system achieve lower sample temperatures.

4. Vacuum jacket: Most ST systems include a vacuum jacket that surrounds the cold finger cryostat. Vacuum jackets are available in a variety of configurations, including optical, compact, and tubular. (The ST-400 cryostat for UHV is generally not equipped with a vacuum jacket.)

5. Optional temperature controller: Many types of automatic temperature controllers are available for use with the ST system. These controllers allow the user to select a desired control temperature while adjusting several other control parameters, including proportional and integral values. The temperature controller then maintains the desired temperature by applying power to the control heater. Controllers purchased through Janis Research include cabling and are tested with the cryostat.

SECTION 3

INSTALLATION

3.1 MOUNTING

The ST system is designed to operate in any orientation. (Greatest efficiency is achieved when operated in the vertical position.) Most ST systems include tapped holes on the base of the vacuum jacket, which can be used for mounting to an optical table. Compact systems, such as the ST-300 and ST-300T include additional mounting holes on the instrumentation skirt.

3.2 ELECTRICAL CONNECTIONS

If an optional automatic temperature controller has been supplied with the ST system, refer to the accompanying controller manual and connect to the appropriate AC outlet. Automatic temperature controllers operate by using a feedback control loop, in which the controller sends output to the control heater based upon the signal from the control thermometer. For this reason, it is important that the control heater and thermometer both be attached to the same copper block, to prevent temperature oscillations. When thermometers are supplied at both the cold finger (for control) and the sample holder (for monitoring the sample temperature), be sure the cold finger thermometer is designated as the control channel. In most cases, the control thermometer will be assigned to Channel A of the controller. Refer to the accompanying wiring diagram at the end of this manual for specific sensor and wiring details.

If no temperature controller has been supplied with the system, refer to the appendix for pin assignments of all feedthroughs installed on the dewar. Mating connectors are supplied which allow the user to attach cables as needed.

SECTION 4

OPERATION

4.1 REMOVING THE VACUUM SHROUD

Before removing the vacuum shroud, vent the vacuum space by turning the shroud evacuation valve knob counter-clockwise. Next, remove the clamp located just below the evacuation valve, and carefully lift the cryostat out of the vacuum shroud. Finally, remove the radiation shield mounting screws and lift the radiation shield off the cold finger. The sample mount and sample holder are now accessible.

4.2 SAMPLE MOUNTING

Most ST systems are supplied with a sample holder. If the sample holder is removed, a thin film of thermal grease (such as Crycon) or thin indium foil should be used to enhance thermal contact when reinstalled. Grease or indium can also be used to improve the thermal contact between the sample and sample holder.

Janis ST systems include provisions for additional electrical feedthroughs for customer wiring of the samples. Small gauge wires (32AWG) should be used to minimize heat leak into the sample, and the wires should be thermally anchored in several spots to the cold finger by using Stycast epoxy, varnish, by tying with nylon string or floss, or by using mylar or aluminum tape.

Once the sample is mounted to the system, reinstall the radiation shield and vacuum shroud. Any visible dirt or lint on the sealing gasket is sufficient to cause a vacuum leak, so be sure the gasket and flanges are clean and lightly greased before mounting the vacuum shroud.

4.3 EVACUATION

Janis ST systems must be evacuated for proper performance. The system includes two independent vacuum spaces. One vacuum space is used to insulate the high efficiency transfer line, while the other vacuum space surrounds the sample region. Both spaces are evacuated prior to shipment from Janis, but it is recommended that both be re-evacuated prior to use. Both vacuum spaces utilize a low leak rate evacuation valve, which allows evacuation and sealing. Prior to operation, connect a turbomolecular or diffusion pump to the transfer line evacuation valve, (located just above the flow regulator valve), and evacuate to a pressure of 1.0×10^{-4} Torr or less. Better vacuum levels provide greater insulation, resulting in shorter cooldown times and lower final temperatures. A cold trapped mechanical vacuum pump can be used instead; however, this may reduce the transfer efficiency. The rigid leg of the transfer line incorporates an activated charcoal getter, to help maintain good vacuum levels when the leg is inserted into a cryogen storage dewar. For this reason, it is preferable to maintain vacuum in the transfer line at all times, and to never allow helium gas or moist air to enter this vacuum space. (In the event moisture or helium gas accidentally contaminates the transfer line vacuum space, re-evacuate the line for several days before operating again.) The transfer line vacuum valve should be closed prior to the beginning of cooldown.

Next, connect the vacuum pump to the cryostat evacuation valve, and pump out the cryostat insulating vacuum space. The cryostat evacuation valve should also be closed once evacuation is complete, to avoid backstreaming of oil from the vacuum pump into the cryostat. Outgassing and o-ring permeation will cause the pressure to rise slowly over time, therefore periodic re-evacuation will be necessary. Re-evacuation of the cryostat is required whenever a new sample is installed, or when the minimum temperature obtained begins to increase.

NOTE!

Some ST systems are equipped for operation at high temperatures. When operating above 325 K, these cryostats should be evacuated <u>continuously</u> to prevent contamination due to heater outgassing..

4.4 INITIAL COOLDOWN (with liquid helium)

1. For quickest cooldown and best results, attach a mechanical vacuum pump to the LHe venting port of the cryostat, and evacuate this space continuously during the first phase of cooldown. This removes any air or moisture remaining in the line from a previous run and shortens the cooldown time by several minutes.

2. Close the flow regulator valve at the transfer line storage dewar leg, by turning the knurled brass operator knob counter-clockwise. Insert the transfer line cryostat leg into the cryostat o-ring sealed port, and tighten the brass o-ring seal nut to form a gas tight seal.

3. Slowly insert the storage dewar leg of the transfer line into the LHe storage dewar. (For easiest adjustment of the transfer rate, the storage dewar should be equipped with a 0 - 5 psi pressure gauge, a venting valve, and a hose adapter for adding helium gas pressure if necessary.) As the leg enters the storage dewar, open the flow regulator valve about 2 - 3 turns, and monitor the pump pressure or exhaust for a few seconds to confirm that the flow is not restricted. (The pump pressure should increase slightly, and the exhaust of the vacuum pump will typically show traces of oil vapor. The flow rate is quite small during the initial phase of the transfer, until the entire inner line becomes cooled.) Close and open the valve several times during the first few minutes of transfer, to be sure the valve does not freeze shut during cooldown, and that the flow is not restricted. Once the leg is fully inserted into the storage dewar, raise the leg about 1 cm from the storage dewar bottom to minimize the chance of frozen debris entering the line.

4. In general, heat introduced by conduction down the transfer line leg is sufficient to maintain positive pressure in the storage dewar. Adjust the storage dewar vent valve to maintain a storage dewar pressure of 1 - 2 psi. (In some cases, helium gas will need to be added to maintain constant pressure in the storage dewar.)

5. After a short wait (typically 5 - 10 minutes) the sample mount will begin to cool rapidly. At this time, the vacuum pump should be disconnected from the system, and the escaping helium vapor vented into the atmosphere or gas collection system. (Be sure to allow the

pump to reach atmospheric pressure before disconnecting, to avoid introducing air and moisture into the line.)

6. Once the sample mount temperature reaches about 5 K, the flow regulator can be closed, until the sample temperature begins to increase. Open the regulator just enough to cool the sample mount to 4.2 K. For best operating efficiency, it is best to open the regulator valve just enough to maintain the desired minimum temperature.

4.5 INITIAL COOLDOWN (with liquid nitrogen)

Cooldown with LN_2 is very similar to operation with LHe, and the procedure above can be used with the following exceptions.

1. Because LN_2 will freeze when it is pumped, it is best not to initiate transfer with a vacuum pump. Instead, use a storage dewar pressure of about 5 psi, and open the flow regulator valve about 3 turns. It will take 5 - 10 minutes before the sample mount temperature begins to drop.

2. It is often necessary to add gas pressure to the storage dewar, to maintain constant pressure of 5 psi. Either helium or nitrogen gas can be used for this purpose.

3. Once the temperature reaches about 100 K, close the flow valve completely, and wait until the sample temperature stabilizes or begins to increase. As in step 6 above, open the flow regulator just enough to reach 77 K. Too large a flow will cause liquid to collect inside the sample mount, making temperature control at higher temperatures difficult or impossible.

4.6 OPERATION

For operation at 4.2 K (or 78 K with LN_2), the flow regulator valve should be opened just enough to maintain this temperature at the sample mount. The flow rate required is a function of the heat load into the cold finger, and will vary depending on configuration and experimental application.

For operation above 4.2 K (or 78 K with LN_2), use an automatic temperature controller, as described in section 4.7 below. Once the temperature is above 20 K, the flow regulator valve can be partially closed, reducing the LHe consumption significantly. Adjustments in the flow regulator setting can be made at any time, either to increase the cooling power or reduce the cryogen consumption. In general, the smallest flow rate should be chosen that will cool the sample to the desired temperature. For elevated temperatures, choose a setpoint using the automatic temperature controller. The temperature controller will supply power to the control heater, and will stabilize the system at the selected temperature setpoint.

Continuous operation below 4.2 K is achieved by reducing the pressure at the helium vent port with a mechanical vacuum pump. The flow regulator valve can be partially opened, continuously replenishing the LHe inside the cold finger, and resulting in a constant temperature of 2.5 K or less.

In order to reach the lowest possible temperature, the flow valve should be opened several turns, and should remain open for about 5 minutes. This will allow LHe to fill the space inside the cold

finger. Next, the flow regulator valve should be completely closed, and the pressure inside the cold finger region reduced, using a vacuum pump on the LHe vent port. The minimum temperature (typically about 1.5 K) will be maintained until all the LHe is depleted from inside the cold finger, about 8 minutes. The process can be repeated as desired.

4.7 TEMPERATURE CONTROL

Most systems are supplied with an automatic temperature controller, silicon diode thermometer, and 25 ohm control heater. Options include other diode or resistance thermometers, thermocouples, and different heater resistance. The actual configuration of your system thermometry can be found on the wiring diagram at the end of this manual.

Most Janis ST systems operate from 1.5 K to 325 K, though an optional 500 K upper limit is available. Choose a temperature setpoint from within the appropriate range, and enter values for Proportional (P), Integral (I), and Derivative (D) parameters. Some experimentation may be required to optimize these settings for a particular application. In general, when operating at the lowest temperatures, (where the heat capacities are smallest), the (P) value should be low, and the (I) value should be high. Derivative (D) control can usually remain zero throughout the operating range. As the control temperature is increased, larger proportional and smaller integral values can improve temperature stability and response time.

Some controllers include an autotuning function that selects appropriate PID values automatically. This function is most useful only for temperatures above 50 K. For complete discussion of this feature, as well as comprehensive controller operating procedures and specifications, refer to the temperature controller manual.

4.8 CHANGING SAMPLES

Before changing samples, the cold finger should be warmed to room temperature. This can be accomplished in either of two ways.

1. The flow regulator valve can be closed, and the system allowed to warm up for several hours. Use the control thermometer to determine when the system is approaching room temperature.

2. The flow regulator valve can be closed, and the temperature controller set for 295 K. Once the thermometer reaches 295 K, wait until the heater power approaches 0%. The evacuation valve can now be opened and the sample changed as described in sections 4.1 and 4.2. (Dry nitrogen or argon gas can be used to break the vacuum if the sample is particularly sensitive to water vapor.)

WARNING!

The radiation shield will remain cold even after the sample has warmed to room temperature. Use gloves when handling a cold radiation shield to avoid low temperature burns, or wait until it warms to room temperature before handling.

4.9 SYSTEM SHUTDOWN

To shut down the system, simply close the flow regulator valve and turn off the temperature controller. If possible, mount a pressure relief valve on the helium venting port. This will permit any cryogen remaining in the inner line to vent safely to atmosphere, while preventing any air or moisture from entering the cryostat. The storage dewar should then be vented to atmospheric pressure, and the transfer line storage dewar leg removed from the storage dewar.

WARNING!

The transfer line leg will be extremely cold upon removal from the storage dewar. Do not touch the leg with bare hands. Always wear insulating gloves when handling the transfer line after use.

SECTION 5

MAINTENANCE

5.1 SCHEDULED MAINTENANCE

The Janis Supertran system requires no regularly scheduled maintenance. The exterior surfaces of the vacuum jacket can be cleaned with a spray household cleaner periodically as necessary. The transfer line should be evacuated periodically, as discussed in section 4.3.

5.2 UNSCHEDULED MAINTENANCE

Unscheduled maintenance may be occasionally required to repair problems arising during the course of operation. These problems may be related to vacuum leaks or wiring failure.

5.3 VACUUM LEAKS

Condensation on the outside of the vacuum jacket, and inability to reach 4 K are indications of a vacuum problem. If these symptoms appear, re-evacuate the shroud as described in paragraph 4.3. If the symptoms disappear, no further action should be required. If the symptoms remain, or reappear quickly, a vacuum leak may be present. Contact Janis Research to obtain further direction in this case.

5.4 WIRING

Occasionally a heater or thermometer wire may be broken during sample removal or installation. If this occurs, reconnect the broken wire using 60/40 rosin core solder. Be sure to insulate the joint with shrinkable PVC tubing or Teflon insulation.

The heater located at the sample mount is designed to accept the normal output of most temperature controllers. Occasionally, however, a heater may burn out. Replacement heater kits are available from Janis, and include all materials and instructions necessary for replacement.

CRYOSTAT SERIAL NUMBER: 11828

10 PIN FEEDTHROUGH LABELED "A"

LOCATION: INSTRUMENTATION SKIRT

PIN A - POS. CURRENT (I+)

PIN B - POS. VOLTAGE (V+)

PIN C - NEG. CURRENT (I-)

PIN D - NEG. VOLTAGE (V-)

(Pins A–D:) **CALIBRATED TG-120-SD GaAlAs #11310 ON SAMPLE MOUNT (CONTROL SENSOR)**

PIN E -

PIN F -

PIN G -

PIN H -

PIN J -

PIN K -

(Pins J–K:) **50 OHM CARTRIDGE HEATER ON SAMPLE MOUNT**

TYPE "E" THERMOCOUPLE ON SAMPLE MOUNT
WIRED TO VACUUM COUPLING ON INSTRUMENTATION SKIRT

A B C D
1 2 3 4
I+ V+ I- V-
C B R G

O
A B L D
B R G K